#### **MEMORANDUM**

TO: Terry Taylor

DATE: December 22, 2011

Anderson, Mulholland and Associates

FROM: R. Infante

FILE: JA94124

RE:

**Data Validation** 

BMS-IMC, Humacao, PR

**Building 5** 

Accutest Job Number: JA94124

#### **SUMMARY**

Full validation was performed on the data for eleven (11) soil samples, one (1) trip blank, (1) field blank, and one (1) equipment blank analyzed for selected volatile organic compounds using EPA method SW-846 8260B and eleven (11) soil samples and one (1) equipment blank analyzed for alcohols (methanol and isopropyl alcohol) by EPA method SW-846 8015 (DAI). The samples were collected at the BMS-IMC Building 5 Area in Humacao, PR on December 7 and 8, 2011 and submitted to Accutest Laboratories that analyzed and reported the results under delivery group (SDG) JA94124.

The sample results were assessed according to USEPA data validation guidance documents in the following order of precedence: "USEPA Region 2, SOP HW-24, Standard Operating Procedure for the Validation of Organic Data Acquired using SW-846 Method 8260B (August 2009-Revision 2), the USEPA National Functional Guidelines for Low Concentration Organic Data Review (August 2009-Revision 2), the USEPA National Functional Guidelines for Organic Data Review for Low Concentration (SOP HW-13, August 2009-Revision 3) (noted herein as the "primary guidance document"). Also, QC criteria from "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods SW-846 (Final Update III, December 1996)," are utilized. The guidelines were modified to accommodate the non-CLP methodology. The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

In general the data is valid as reported and may be used for decision making purposes. The data results are acceptable for use. Some of the results were qualified.

#### **SAMPLES**

The samples included in the review are listed below

FIELD SAMPLE ID	LABORATORY ID	ANALYSIS
I-12 (9.5 - 10.5)	JA94124-1	VOCs, ALCOHOLS
I-13 (12.5 - 13.5)	JA94124-2	VOCs, ALCOHOLS
I-14 (8.5 - 9.5)	JA94124-3	VOCs, ALCOHOLS
I-15 (5 - 6)	JA94124-4	VOCs, ALCOHOLS
I-16 (6 - 7)	JA94124-5	VOCs, ALCOHOLS
I-17 (10 - 11)	JA94124-6	VOCs, ALCOHOLS
I-18 (7 - 8)	JA94124-7	VOCs, ALCOHOLS
I-19 (8 - 9)	JA94124-8	VOCs, ALCOHOLS
EB120811	JA94124-9	VOCs, ALCOHOLS
P-9 (4.5 - 6)	JA94124-10	VOCs, ALCOHOLS
P-8 (4 - 5)	JA94124-11	VOCs, ALCOHOLS
P-5 (4.5 - 5.5)	JA94124-12	VOCs, ALCOHOLS
FB120811	JA94124-13	VOCs
TB120811	JA94124-14	VOCs

### **REVIEW ELEMENTS**

Sample data were reviewed for the following parameters, where applicable to the method

- o Agreement of analysis conducted with chain of custody (COC) form
- o Holding time and sample preservation
- o Gas chromatography/mass spectrometry (GC/MS) tunes
- o Initial and continuing calibrations
- o Method blanks/trip blanks/field blank
- Surrogate spike recovery
- o Matrix spike/matrix spike duplicate (MS/MSD) results
- o Internal standard performance
- o Field duplicate results
- o Laboratory control sample/laboratory control sample duplicate (LCS/LCSD) results

o Quantitation limits and sample results

#### DISCUSSION

### Agreement of Analysis Conducted with COC Request

Sample reports corresponded to the analytical request designated on the chain-of-custody form.

### **Holding Times and Sample Preservation**

The cooler temperatures were within the QC acceptance criteria of  $4^{\circ}$ C  $\pm$   $2^{\circ}$ C.

Sample preservation was acceptable.

Samples analyzed within method recommended holding time.

#### **GC/MS Tunes**

The frequency and abundance of bromofluorobenzene (BFB) tunes were within the QC acceptance criteria. All samples were analyzed within the tuning criteria associated with the method.

### Initial and Continuing Calibrations

### **VOCs**

The percent relative standard deviations (%RSDs) and response factors (RFs) of all target analytes were within the QC acceptance criteria in the initial calibration. Correlation coefficients (r²) of target analytes were within the QC acceptance criteria. Ongoing accuracy of the instrument was determined by the analysis of a continuing calibration standard. All initial and continuing calibrations met the acceptance criteria except for the following analytes:

DATE	LAB FILE ID#	CRITERIA OUT: %D	COMPOUND	AFFECTED SAMPLES
=======			========	
12/12/11	cc435-20	- 23.6	MIBK	JA94124-9; -13; -14

Qualify results (j) in affected samples.

#### Alcohols

The percent relative standard deviations (%RSDs) and response factors (RFs) of all target analytes were within the QC acceptance criteria in the initial calibration. Correlation coefficients (r²) of target analytes were within the QC acceptance criteria. Ongoing accuracy of the instrument was determined by the analysis of a continuing calibration standard. All initial and continuing calibrations met the acceptance criteria

### Method Blank/Trip Blank/Field Blank

Target analytes were not detected in laboratory method blanks for VOCs and alcohols.

No target analytes (VOCs) in the trip/field/equipment blanks associated with this data set.

No target analytes (ALCOHOLS) in the equipment blank associated with this data set. No trip/field blanks associated with this data set were analyzed for alcohols.

### **Surrogate Spike Recovery**

The surrogate recoveries were within the laboratory QC acceptance limits in all samples analyzed for VOCs and alcohols except for the followings:

- JA94124-6 for Hexanol: Outside control limits due to matrix interference. Confirmed by MS/MSD.
- JA94124-6MS for Hexanol: Outside control limits due to matrix interference.
- JA94124-6MSD for Hexanol: Outside control limits due to matrix interference

No action taken; surrogates recoveries within control limits in signal #2,

### MS/MSD

#### **VOCs**

Matrix spike was performed on samples JA93776-3MS/-3MSD (Aqueous); JA94124-5MS/-5MSD (Soil); and JA94124-3MS/-3MSD. Recoveries and RPD for the MS/MSD were within laboratory control limits except for the followings:

MS OR MSD	COMPOUND	% R	RPD	QC LIMITS	ACTION
JA94124-5					
MS	Ethylbenzene	172		20144	Qualify_results_(J)_
MS	Toluene	146		29138	affected_samples
MS	Xylenes(Total)	176		18145	
A CC					

## Affected samples: JA94124-2; -3; -4; -5; -7; -8; -11; -12

### <u>Alcohols</u>

Matrix spike was performed on samples JA93907-1MS/1MSD (Aqueous) and JA93968-1MS/-1MSD (Soil). Recoveries and RPD for the MS/MSD were within laboratory control limits.

### **Internal Standard Performance**

### **VOCs**

Samples were spiked with the method specified internal standard. Internal standard performance met the QC acceptance criteria in all sample analyses.

### **Laboratory/Field Duplicate Results**

Laboratory duplicate associated with data package were samples JA94124-4 (VOCs). RPD results were within laboratory and generally acceptable control limits except for the followings:

COMPOUND	SQL	SAMPLE CONC.	DUPLICATE CONC.	RPD	ACTION
Benzene	0.14	0.76	0.31	84	Qualify results (J) in
Ethylbenzene	0.15	0.38	ND	200	sample JA94124-4
Xylene(total)	0.19	7.5	2.0	116	

Note: laboratory states that: "High RPD due to possible sample analyzed from different vials"; therefore results qualified based on professional judgment

No field/laboratory duplicates were analyzed for alcohols in this data set. MS/MSD results used to assess precision. RPD results were within laboratory and generally acceptable control limits.

### LCS/LCSD Results

### **VOCs**

The laboratory analyzed one LCS (blank spike) associated with each matrix from this data set. The % recoveries of all spiked analytes were within the laboratory QC acceptance limits.

### <u>Alcohols</u>

The laboratory analyzed one LCS (blank spike) associated with each matrix from this data set. The % recoveries of all spiked analytes were within the laboratory QC acceptance limits.

### **Quantitation Limits and Sample Results**

Dilutions were not required with this data set except for the following samples (alcohols):

SAMPLE ID	DILUTION FACTOR	REASON FOR DILUTION
JA94124-6	10 x	Ethylbenzene and total xylenes outside calibration range
JA94124-10	10 x	MIBK and total xylenes outside calibration range

Calculations were spot checked.

More than 40 % RPD for detected Isopropyl Alcohol concentrations between the two GC columns in samples JA94124-1. Results qualified as estimated (J).

### Certification

The following samples JA94124-1; JA94124-2; JA94124-3; JA94124-4; JA94124-5; JA94124-6; JA94124-7; JA94124-8; JA94124-9; JA94124-10; JA94124-11; JA94124-12; JA94124-13; and JA94124-14 were analyzed following standard procedures accepted by regulatory agencies. The quality control requirements met the methods criteria except in the occasions described in this document. The results are valid.

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Rafael Infante

Chemist License 1888

Client Sample ID: I-12(9.5-10.5)

Lab Sample ID: Matrix:

JA94124-1

SO - Soil

Method:

Project:

SW846 8260B SW846 5035

BMS-ICM, Humacao, PR

Date Sampled: 12/07/11 Date Received: 12/09/11

Percent Solids: 87.4

	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
Run #1	E185931.D	1	12/14/11	OTR	12/09/11 13:00	n/a	VE8180
Run #2	E185929.D	1	12/14/11	OTR	12/09/11 13:00	n/a	VE8180

	Initial Weight	Final Volume	Methanol Aliquot
Run #1	5.4 g	5.0 ml	2.0 ul
Run #2	5.4 g	5.0 ml	100 ul

### **VOA Special List**

CAS No.	Compound	Result	RL	MDL	Units	Q
67-64-1 71-43-2 100-41-4 108-10-1	Acetone Benzene Ethylbenzene 4-Methyl-2-pentanone(MIBK)	69800 ND <sup>a</sup> 361000 127000	30000 60 3000 15000	20000 8.0 450 7900	ug/kg ug/kg ug/kg ug/kg	
108-88-3 1330-20-7	Toluene Xylene (total)	1060 a 1270000	60 3000	23 550	ug/kg ug/kg	
CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limi	its	
1868-53-7 17060-07-0 2037-26-5 460-00-4	Dibromofluoromethane 1,2-Dichloroethane-D4 Toluene-D8 4-Bromofluorobenzene	93% 98% 92% 86%	91% 97% 90% 80%	67-1: 66-1: 76-1: 53-1-	30% 25%	

(a) Result is from Run# 2



ND = Not detected

MDL - Method Detection Limit

RL = Reporting Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank

 Client Sample ID:
 I-12(9.5-10.5)

 Lab Sample ID:
 JA94124-1

 Matrix:
 SO - Soil

 Matrix:
 SO - Soil

 Method:
 SW846-8015 (DAI)

Project:

Date Sampled: 12/07/11 Date Received: 12/09/11 Percent Solids: 87.4

BMS-ICM, Humacao, PR

	File ID	DF	Analyzed	By	<b>Prep Date</b>	Prep Batch	<b>Analytical Batch</b>
Run #1	GH85804.D	1	12/13/11	XPL	n/a	n/a	GGH3921
l- "-							

Run #2

Initial Weight
Run #1 5.0 g

Run #2

CAS No.	Compound	Result	RL	MDL	Units	Q
67-63-0 67-56-1	Isopropyl Alcohol <sup>a</sup> Methanol	40100 ND	110 230	44 59	ug/kg ug/kg	
CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Lim	its	
111-27-3	Hexanol	128%		58-1	37%	
111-27-3	Hexanol	102%		58-1	37%	

(a) More than 40 % RPD for detected concentrations between the two GC columns.



ND = Not detected

MDL - Method Detection Limit

J = Indicates an estimated value

RL = Reporting Limit

E = Indicates value exceeds calibration range

B = Indicates analyte found in associated method blank N = Indicates presumptive evidence of a compound

Page 1 of 1

Client Sample ID: I-13(12.5-13.5) Lab Sample ID:

Matrix:

Method:

JA94124-2

SO - Soil SW846 8260B SW846 5035 **Date Sampled:** 12/07/11 Date Received: 12/09/11

Percent Solids: 81.6

By

RS

Project: BMS-ICM, Humacao, PR

File ID Y118377.D Run #1

DF 1

Analyzed 12/10/11

**Prep Date** 12/09/11 13:00

**Prep Batch** n/a

**Analytical Batch** VY5057

Run #2

**Initial Weight** 

5.3 g

Run #1

Run #2

### VOA Special List

CAS No.	Compound	Result	RL	MDL	Units	Q
67-64-1 71-43-2 100-41-4 108-10-1 108-88-3 1330-20-7	Acetone Benzene Ethylbenzene 4-Methyl-2-pentanone(MIBK) Toluene Xylene (total)	20.1 1.6 0.31 J ND ND J 11.3 J	12 1.2 1.2 5.8 1.2	7.7 0.15 0.17 3.0 0.44 0.21	ug/kg ug/kg ug/kg ug/kg ug/kg ug/kg	J
CACAT.			D ".			

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
1868-53-7	Dibromofluoromethane	90%		67-131%
17060-07-0	1,2-Dichloroethane-D4	86%		66-130%
2037-26-5	Toluene-D8	96%		76-125%
460-00-4	4-Bromofluorobenzene	93%		53-142%



ND = Not detected

MDL - Method Detection Limit

RL = Reporting Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank

Page 1 of 1

Client Sample ID: I-13(12.5-13.5) Lab Sample ID:

JA94124-2

SO - Soil

SW846-8015 (DAI)

BMS-ICM, Humacao, PR

**Date Sampled:** 12/07/11

Date Received: 12/09/11

Percent Solids: 81.6

Run #1

Matrix:

Method:

Project:

File ID GH85791.D DF 1

Analyzed 12/13/11

By XPL n/a

**Prep Date** 

Limits

58-137%

58-137%

**Prep Batch** n/a

Q

**Analytical Batch** GGH3921

Run #2

**Initial Weight** 

Compound

Methanol

Isopropyl Alcohol

5.0 g

Run #1

Run #2

CAS No.

67-63-0

67-56-1

Result	RL	MDL	Units
ND	120	47	ug/kg
731	250	63	ug/kg

CAS No.	Surrogate Recoveries	Run# 1	Run# 2
111-27-3	Hexanol	103%	

Hexanol 103% 111-27-3 103% Hexanol



ND = Not detected

MDL - Method Detection Limit

RL = Reporting Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank

By

RS

**Client Sample ID:** I-14(8.5-9.5) Lab Sample ID: JA94124-3

Matrix:

SO - Soil

**Date Sampled:** 12/07/11 Date Received: 12/09/11

Method:

SW846 8260B SW846 5035

Project:

BMS-ICM, Humacao, PR

DF

1

Percent Solids: 79.3

Run #1

File ID Y118374.D Analyzed 12/10/11

**Prep Date** 12/09/11 13:00

**Prep Batch** n/a

**Analytical Batch** VY5057

Run #2

**Initial Weight** 

Run #1 5.3 g

Run #2

**VOA Special List** 

CAS No.	Compound	Result	RL	MDL	Units	Q
67-64-1 71-43-2 100-41-4 108-10-1 108-88-3	Acetone Benzene Ethylbenzene 4-Methyl-2-pentanone(MIBK) Toluene	12.0 0.42 32.5 3 ND ND J	12 1.2 1.2 5.9 1.2	7.9 0.16 0.18 3.1 0.45	ug/kg ug/kg ug/kg ug/kg ug/kg	J
1330-20-7	Xylene (total)	82.6 <b>J</b>	1.2	0.22	ug/kg	

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
1868-53-7	Dibromofluoromethane	93%		67-131%
17060-07-0	1,2-Dichloroethane-D4	94%		66-130%
2037-26-5	Toluene-D8	97%		76-125%
460-00-4	4-Bromofluorobenzene	91%		53-142%



ND = Not detected

MDL - Method Detection Limit

RL = Reporting Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank N = Indicates presumptive evidence of a compound

**Client Sample ID:** I-14(8.5-9.5) Lab Sample ID: JA94124-3 Matrix:

SO - Soil

SW846-8015 (DAI)

**Date Sampled:** 12/07/11 Date Received: 12/09/11

Percent Solids: 79.3

Project: BMS-ICM, Humacao, PR

By File ID DF **Analyzed Prep Date Prep Batch Analytical Batch** GH85797.D 12/13/11 XPL GGH3921 Run #1 1 n/a n/a

Run #2

Method:

**Initial Weight** Run #1 5.0 g

Run #2

CAS No. RL MDL Compound Result Units Q 67-63-0 Isopropyl Alcohol ND 130 48 ug/kg 67-56-1 Methanol 65 ND 250 ug/kg CAS No. **Surrogate Recoveries** Run# 1 Run# 2 Limits 111-27-3 Hexanol 105% 58-137% 111-27-3 Hexanol 58-137% 88%



ND = Not detected

MDL - Method Detection Limit

RL = Reporting Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank

Page 1 of 1

Client Sample ID: I-15(5-6)

Lab Sample ID: Matrix:

JA94124-4

SO - Soil

SW846 8260B SW846 5035

Date Received: 12/09/11 Percent Solids: 83.1

**Date Sampled:** 12/07/11

Method: Project: BMS-ICM, Humacao, PR

File ID Run #1 Y118375.D DF 1

By Analyzed 12/10/11 RS **Prep Date** 12/09/11 13:00

Prep Batch n/a

**Analytical Batch** 

VY5057

Run #2

**Initial Weight** 

Run #1 5.9 g

Run #2

### **VOA Special List**

CAS No.	Compound	Result	RL	MDL	Units	Q
67-64-1 71-43-2 100-41-4 108-10-1 108-88-3 1330-20-7	4-Methyl-2-pentanone(MIBK)	7.8 0.76 0.38 J ND ND J 7.5 J	10 1.0 1.0 5.1 1.0	6.8 0.14 0.15 2.7 0.39 0.19	ug/kg ug/kg ug/kg ug/kg ug/kg ug/kg	J J

CAS No. Sur	rogate Recoveries	Run# 1	Run# 2	Limits
17060-07-0 1,2- 2037-26-5 Tolu	romofluoromethane Dichloroethane-D4 tene-D8 romofluorobenzene	91% 93% 97% 93%		67-131% 66-130% 76-125% 53-142%



ND = Not detected

MDL - Method Detection Limit

RL = Reporting Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank N = Indicates presumptive evidence of a compound

Page 1 of 1

Client Sample ID: I-15(5-6)

Lab Sample ID: Matrix:

JA94124-4 SO - Soil

Method: Project:

SW846-8015 (DAI) BMS-ICM, Humacao, PR

DF

1

**Date Sampled:** 12/07/11

Date Received: 12/09/11

Percent Solids: 83.1

Run #1

File ID GH85798.D Analyzed 12/13/11

By XPL

**Prep Date** n/a

**Prep Batch** 

Q

**Analytical Batch** 

GGH3921 n/a

Run #2

**Initial Weight** Run #1 5.1 g

Run #2

CAS No.

111-27-3

111-27-3

Result RL**MDL** Units

67-63-0 Isopropyl Alcohol 67-56-1 Methanol

Compound

Hexanol

Hexanol

ND 120 45 ND 240 61

ug/kg ug/kg

CAS No. **Surrogate Recoveries** Run#1

Run# 2 109%

106%

58-137% 58-137%

Limits

BHE ASOCIADO O tael Infante Viende/ 1888

ND = Not detected

Client Sample ID: I-16(6-7)

Lab Sample ID:

JA94124-5

Matrix: Method: SO - Soil SW846 8260B SW846 5035

Project:

BMS-ICM, Humacao, PR

5.0 ml

Date Sampled: 12/07/11 Date Received: 12/09/11

Percent Solids: 86.6

File ID E185935.D Run #1 a

DF 1

Analyzed 12/14/11

By OTR

**Prep Date** 12/09/11 13:00

**Prep Batch** n/a

**Analytical Batch** VE8180

Run #2

**Initial Weight** 5.9 g

**Final Volume Methanol Aliquot** 100 ul

Run #1 Run #2

**VOA Special List** 

CAS No.	Compound	Result	RL	MDL	Units	Q
67-64-1 71-43-2 100-41-4 108-10-1 108-88-3 1330-20-7	Acetone Benzene Ethylbenzene 4-Methyl-2-pentanone(MIBK) Toluene Xylene (total)	ND ND 1840 <b>J</b> 249 ND <b>J</b> 6040 <b>J</b>	570 57 57 280 57	380 7.5 8.4 150 21	ug/kg ug/kg ug/kg ug/kg ug/kg ug/kg	J

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
1868-53-7	Dibromofluoromethane	92%		67-131%
17060-07-0	1,2-Dichloroethane-D4	97%		66-130%
2037-26-5	Toluene-D8	95%		76-125%
460-00-4	4-Bromofluorobenzene	84%		53-142%

(a) Diluted due to high concentration of target compound.





MDL - Method Detection Limit

RL = Reporting Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank

Client Sample ID: I-16(6-7) Lab Sample ID:

Matrix:

Method:

Project:

JA94124-5 SO - Soil

SW846-8015 (DAI)

BMS-ICM, Humacao, PR

Date Sampled: 12/07/11

Date Received: 12/09/11 Percent Solids: 86.6

File ID Run #1 GH85799.D Run #2

DF 1

Analyzed By 12/13/11 XPL

**Prep Date** n/a

**Prep Batch** n/a

Q

**Analytical Batch** GGH3921

Initial Weight 5.0 g

Run #1

CAS No.

111-27-3

111-27-3

Run #2

Result RL MDL Units

Run# 2

67-63-0 Isopropyl Alcohol 67-56-1 Methanol

Hexanol

Hexanol

Compound

ND 120 ND 230 44 59 ug/kg ug/kg

CAS No. **Surrogate Recoveries** Run# 1

105% 103%

58-137% 58-137%

Limits

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ND = Not detected

MDL - Method Detection Limit

RL = Reporting Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank

Client Sample ID: I-17(10-11) Lab Sample ID:

JA94124-6

**Date Sampled:** 12/08/11

Date Received: 12/09/11

Matrix: Method: SO - Soil SW846 8260B SW846 5035

Percent Solids: 90.7

Project:

BMS-ICM, Humacao, PR

	File ID	DF	Analyzed	Ву	Prep Date	Prep Batch	Analytical Batch
Run #1	E185928.D	1	12/14/11	OTR	12/09/11 13:00	n/a	VE8180
Run #2	E185932.D	10	12/14/11	OTR	12/09/11 13:00	n/a	VE8180

	Initial Weight	Final Volume	Methanol Aliquot
Run #1	5.0 g	5.0 ml	2.0 ul
Run #2	5.0 g	5.0 ml	1.0 ul

RL

MDL

Units

Q

### **VOA Special List**

Compound

CAS No.

67-64-1	Acetone	ND	30000	20000	ug/kg
71-43-2	Benzene	ND	3000	400	ug/kg
100-41-4	Ethylbenzene	1710000 a	60000	8900	ug/kg
108-10-1	4-Methyl-2-pentanone(MIBK)	34900	15000	7900	ug/kg
108-88-3	Toluene	4420	3000	1100	ug/kg
1330-20-7	Xylene (total)	5550000 a	60000	11000	ug/kg
CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limi	its
CAS No. 1868-53-7	Surrogate Recoveries  Dibromofluoromethane	<b>Run# 1</b> 93%	<b>Run# 2</b> 93%	<b>Limi</b> 67-13	
	•				31%
1868-53-7	Dibromofluoromethane	93%	93%	67-13	31% 30%

Result

(a) Result is from Run# 2



ND = Not detected

MDL - Method Detection Limit

RL = Reporting Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank N = Indicates presumptive evidence of a compound



Client Sample ID: I-17(10-11) Lab Sample ID: JA94124-6

Matrix:

SO - Soil

Method: SW846-8015 (DAI) Project:

BMS-ICM, Humacao, PR

Date Sampled: 12/08/11 Date Received: 12/09/11

Percent Solids: 90.7

**Prep Date Analytical Batch** File ID DF Analyzed By **Prep Batch** GH85792.D 12/13/11 XPL GGH3921 Run #1 1 n/a n/a

Run #2

Initial Weight

Hexanol

Run #1 5.1 g

Run #2

CAS No. Compound Result RL **MDL** Units Q ug/kg 67-63-0 Isopropyl Alcohol 1220 110 41 67-56-1 Methanol 301 220 56 ug/kg CAS No. **Surrogate Recoveries** Run#1 Run# 2 Limits 143% a 111-27-3 Hexanol 58-137% 111-27-3 111%

(a) Outside control limits due to matrix interference. Confirmed by MS/MSD.



58-137%

N = Indicates presumptive evidence of a compound

Page 1 of 1

Client Sample ID: I-18(7-8)

Lab Sample ID:

JA94124-7

SO - Soil

Matrix: Method:

SW846 8260B SW846 5035

Date Received:

**Date Sampled:** 12/08/11 12/09/11

Percent Solids: 85.6

Project:

BMS-ICM, Humacao, PR

DF

1

File ID Y118378.D Run #1

Analyzed By 12/10/11 RS **Prep Date** 

**Prep Batch** n/a

**Analytical Batch** VY5057

12/09/11 13:00

Run #2

**Initial Weight** 

Run #1 5.5 g

Run #2

### **VOA Special List**

CAS No.	Compound	Result	RL	MDL	Units	Q
67-64-1 71-43-2 100-41-4 108-10-1 108-88-3 1330-20-7	Acetone Benzene Ethylbenzene 4-Methyl-2-pentanone(MIBK) Toluene Xylene (total)	7.7 0.31 1.7 ND ND 11.9	11 1.1 1.1 <b>7</b> 5.3 1.1 <b>7</b> 1.1 <b>J</b>	7.0 0.14 0.16 2.8 0.40 0.20	ug/kg ug/kg ug/kg ug/kg ug/kg ug/kg	J

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
1868-53-7	Dibromofluoromethane	91%		67-131%
17060-07-0	1,2-Dichloroethane-D4	92%		66-130%
2037-26-5	Toluene-D8	96%		76-125%
460-00-4	4-Bromofluorobenzene	91%		53-142%



ND = Not detected

MDL - Method Detection Limit

RL = Reporting Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank

Client Sample ID: I-18(7-8)

Lab Sample ID: Matrix:

JA94124-7 SO - Soil

Method:

SW846-8015 (DAI)

Project:

BMS-ICM, Humacao, PR

**Date Sampled:** 12/08/11 Date Received: 12/09/11

Percent Solids: 85.6

Run #1

File ID GH85800.D DF 1

Analyzed 12/13/11

**Prep Date** n/a

**Prep Batch** 

Q

**Analytical Batch** 

n/a

GGH3921

Run #2

**Initial Weight** 

Compound

Run #1

Run #2

CAS No.

Result RL **MDL** 

By

XPL

67-63-0 Isopropyl Alcohol 67-56-1 Methanol

5.2 g

ND 110 ND 220 43 58

ug/kg ug/kg

Units

CAS No. **Surrogate Recoveries** Run# 1 Run# 2 Limits

111-27-3 Hexanol 58-137% 114% 111-27-3 Hexanol 107% 58-137%



ND = Not detected

MDL - Method Detection Limit

RL = Reporting Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank

By

RS

Page 1 of 1

Client Sample ID: I-19(8-9)

Lab Sample ID:

JA94124-8

SO - Soil

Matrix: Method:

SW846 8260B SW846 5035

Project:

BMS-ICM, Humacao, PR

**Date Sampled:** 12/08/11 Date Received: 12/09/11

Percent Solids: 84.6

File ID Y118379.D Run #1

DF 1

**Analyzed** 12/10/11

**Prep Date** 

**Prep Batch** 12/09/11 13:00 n/a

VY5057

**Analytical Batch** 

Run #2

**Initial Weight** 

5.6 g

Run #1

Run #2

CAS No.

**VOA Special List** 

CAS No.	Compound	Result	RL	MDL	Units	Q
67-64-1	Acetone	13.2	11	7.0	ug/kg	
71-43-2	Benzene	0.51	1.1	0.14	ug/kg	J
100-41-4	Ethylbenzene	7.8	1.1	0.16	ug/kg	
108-10-1	4-Methyl-2-pentanone(MIBK)	ND	5.3	2.8	ug/kg	
108-88-3	Toluene	ND	1.1	0.40	ug/kg	
1330-20-7	Xylene (total)	23.2	1.1	0.19	ug/kg	

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
1868-53-7	Dibromofluoromethane	90%		67-131%
17060-07-0	1,2-Dichloroethane-D4	88%		66-130%
2037-26-5	Toluene-D8	96%		76-125%
460-00-4	4-Bromofluorobenzene	92%		53-142%



Client Sample ID: I-19(8-9)

Lab Sample ID:

JA94124-8

Matrix: Method: Project:

SO - Soil

SW846-8015 (DAI)

BMS-ICM, Humacao, PR

**Date Sampled:** 12/08/11 Date Received: 12/09/11

Percent Solids: 84.6

Run #1

File ID GH85801.D DF 1

Analyzed By XPL

12/13/11

**Prep Date** n/a

**Prep Batch** n/a

Q

**Analytical Batch** GGH3921

Run #2

**Initial Weight** 

Run #1

Run #2

5.1 g

CAS No.	Compound	Result	RL	MDL	Units
67-63-0 67-56-1	Isopropyl Alcohol Methanol	ND ND	120 230	44 60	ug/kg ug/kg
61637		" -			

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
111-27-3	Hexanol	104%		58-137%
111-27-3	Hexanol	101%		58-137%



Page 1 of 1

Client Sample ID: EB120811

Lab Sample ID:

JA94124-9 AQ - Equipment Blank

Matrix: Method: Project:

SW846 8260B

BMS-ICM, Humacao, PR

**Date Sampled:** 12/08/11 Date Received: 12/09/11

Percent Solids: n/a

**Analytical Batch Prep Date Prep Batch** File ID DF Analyzed By Run #1 4B13608.D 12/12/11 RS V4B588 1 n/a n/a

Run #2

**Purge Volume** 

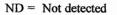
Run #1 5.0 ml

Run #2

### **VOA Special List**

CAS No.	Compound	Result	RL	MDL	Units	Q
67-64-1	Acetone	ND	10	7.6	ug/l	
71-43-2	Benzene	ND	1.0	0.22	ug/l	
100-41-4	Ethylbenzene	ND _	1.0	0.21	ug/l	
108-10-1	4-Methyl-2-pentanone(MIBK)	ND $\mathcal{J}$	5.0	1.2	ug/l	
108-88-3	Toluene	ND	1.0	0.15	ug/l	
1330-20-7	Xylene (total)	ND	1.0	0.17	ug/l	
CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limit	ts	
10/0 50 5						
1868-53-7	Dibromofluoromethane	96%		77-12	:0%	
1868-53-7 17060-07-0	Dibromofluoromethane 1,2-Dichloroethane-D4	96% 90%		77-12 70-12		
					7%	
17060-07-0	1,2-Dichloroethane-D4	90%		70-12	7%	ASOCIADO DE
17060-07-0 2037 <b>-</b> 26-5	1,2-Dichloroethane-D4 Toluene-D8	90% 93%		70-12 79-12	7%	ASOCIADO DE

MDL - Method Detection Limit



RL = Reporting Limit E = Indicates value exceeds calibration range J = Indicates an estimated value

B = Indicates analyte found in associated method blank

N = Indicates presumptive evidence of a compound

Mende/ 10 1888 Client Sample ID: EB120811 Lab Sample ID: JA94124-9

Matrix:

JA94124-9

Method: Project: AQ - Equipment Blank SW846-8015 (DAI) BMS-ICM, Humacao, PR **Date Sampled:** 12/08/11 **Date Received:** 12/09/11

Percent Solids: n/a

Q

	File ID	DF	Analyzed	Ву	Prep Date	Prep Batch	Analytical Batch
Run #1	GH85784.D	1	12/13/11	XPL	n/a	n/a	GGH3920
Run #2							

CAS No.	Compound	Result	RL	MDL	Units	
67-63-0 67-56-1	Isopropyl Alcohol Methanol	ND ND	100 200	30 46	ug/l ug/l	
CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Lim	its	
111-27-3	Hexanol	103%		48-1	50%	
111-27-3	Hexanol	104%			50%	



E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank N = Indicates presumptive evidence of a compound

Client Sample ID: P-9(4.5-6) Lab Sample ID: JA94124-10

Matrix:

Method:

Project:

SO - Soil

SW846 8260B SW846 5035

BMS-ICM, Humacao, PR

**Date Sampled:** 12/08/11 **Date Received:** 12/09/11

Percent Solids: 87.5

	File ID	DF	Analyzed	Ву	Prep Date	Prep Batch	Analytical Batch
Run #1	E185930.D	1	12/14/11	OTR	12/09/11 13:00	n/a	VE8180
Run #2	E185934.D	1	12/14/11	OTR	12/09/11 13:00	n/a	VE8180
Run #3	E185936.D	10	12/14/11	OTR	12/09/11 13:00	n/a	VE8180

	Initial Weight	Final Volume	Methanol Aliquot
Run #1	5.9 g	5.0 ml	100 ul
Run #2	5.9 g	5.0 ml	2.0 ul
Run #3	5.9 g	5.0 ml	4.0 ul

### **VOA Special List**

CAS No.	Compound	Result	RL	MDL	Units	Q
67-64-1	Acetone	25700 a	28000	18000	ug/kg	
71-43-2 100-41-4	Benzene Ethylbenzene	46.2 488000 a	56 2800	7.4 410	ug/kg ug/kg	
108-10-1 108-88-3	4-Methyl-2-pentanone(MIBK) Toluene	850000 <sup>b</sup> 1840	69000 56	37000 21	ug/kg ug/kg	
1330-20-7	Xylene (total)	1750000 b	14000	2600	ug/kg	
CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Run	<b>#</b> 3	Limits
1868-53-7	Dibromofluoromethane	91%	92%	92%		67-131%
17060-07-0 2037-26-5	1,2-Dichloroethane-D4 Toluene-D8	97% 87%	96% 92%	98% 91%		66-130% 76-125%
460-00-4	4-Bromofluorobenzene	81%	85%	86%		53-142%



(b) Result is from Run# 3



ND = Not detected

MDL - Method Detection Limit

RL = Reporting Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank

By

XPL

Page 1 of 1

Client Sample ID: P-9(4.5-6) Lab Sample ID:

Matrix:

JA94124-10 SO - Soil

Method:

SW846-8015 (DAI)

Project:

BMS-ICM, Humacao, PR

**Date Sampled:** 12/08/11 Date Received: 12/09/11

Percent Solids: 87.5

Run #1

File ID GH85805.D DF 1

Analyzed 12/13/11

**Prep Date** n/a

**Prep Batch** n/a

Q

**Analytical Batch** GGH3921

Run #2

**Initial Weight** 

Compound

Run #1

CAS No.

111-27-3

111-27-3

Run #2

Result  $\mathbf{RL}$ **MDL** Units

67-63-0 Isopropyl Alcohol 67-56-1 Methanol

5.0 g

7390 2680

Run# 1

110 44 230 59

Run# 2

ug/kg ug/kg

CAS No. **Surrogate Recoveries** 

Hexanol

Hexanol

95% 96%

58-137% 58-137%

Limits

tael Infante Mende/ 1888

ND = Not detected

MDL - Method Detection Limit

RL = Reporting Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank

Client Sample ID: P-8(4-5)

Lab Sample ID:

JA94124-11

Matrix:

SO - Soil

Method:

SW846 8260B SW846 5035

Project:

BMS-ICM, Humacao, PR

**Date Sampled:** 12/08/11 Date Received: 12/09/11

Percent Solids: 81.3

File ID Run #1 Y118380.D DF 1

Analyzed By 12/10/11 RS **Prep Date** 12/09/11 13:00

**Prep Batch** n/a

**Analytical Batch** VY5057

Run #2

**Initial Weight** 

5.7 g Run #1

Run #2

**VOA Special List** 

CAS No.	Compound	Result	RL	MDL	Units Q
67-64-1	Acetone	ND	11	7.1	ug/kg
71-43-2	Benzene	ND	1.1	0.14	ug/kg
100-41-4	Ethylbenzene	ND J	1.1	0.16	ug/kg
108-10-1	4-Methyl-2-pentanone(MIBK)	ND	5.4	2.8	ug/kg
108-88-3	Toluene	ND J	1.1	0.41	ug/kg
1330-20-7	Xylene (total)	ND 🤰	1.1	0.20	ug/kg
CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limi	its
1868-53-7	Dibromofluoromethane	94%		67-1	31%
17060-07-0	1,2-Dichloroethane-D4	98%		66-1	30%
2037-26-5	Toluene-D8	98%		76-1	25861400 A
460-00-4	4-Bromofluorobenzene	91%		53,	A POINT OF PLAN
				,	31% 30% 258CIADO DE PRINCIPAL D
					1 1888 S
				111	No. JCHO



MDL - Method Detection Limit

RL = Reporting Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank

Client Sample ID: P-8(4-5) Lab Sample ID:

Matrix:

JA94124-11 SO - Soil

Method:

SW846-8015 (DAI)

Project:

BMS-ICM, Humacao, PR

**Date Sampled:** 12/08/11 Date Received: 12/09/11

Percent Solids: 81.3

**Prep Date Analytical Batch** File ID DF Analyzed  $\mathbf{B}\mathbf{y}$ **Prep Batch** GH85806.D 12/13/11 XPL GGH3921 Run #1 1 n/a n/a

Run #2

**Initial Weight** 

Run #1 5.1 g

Run #2

CAS No.	Compound	Result	RL	MDL	Units	Q
67-63-0 67-56-1	Isopropyl Alcohol Methanol	ND 257	120 240	46 62	ug/kg ug/kg	
CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Lim	iits	
111-27-3	Hexanol Hexanol	83% 85%			3 <b>7</b> %	



ND = Not detected

MDL - Method Detection Limit

RL = Reporting Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank

**Client Sample ID:** P-5(4.5-5.5) Lab Sample ID: JA94124-12

Matrix: Method:

Project:

SO - Soil

SW846 8260B SW846 5035 BMS-ICM, Humacao, PR

**Date Sampled:** 12/08/11 Date Received: 12/09/11

Percent Solids: 83.9

	File ID	DF	Analyzed	Ву	Prep Date	Prep Batch	Analytical Batch
Run #1	Y118381.D	1	12/10/11	RS	12/09/11 13:00	n/a	VY5057
Run #2	E185933.D	1	12/14/11	OTR	12/09/11 13:00	n/a	VE8180

	Initial Weight	Final Volume	Methanol Aliquot
Run #1	5.8 g		
Run #2	5.9 g	5.0 ml	100 ul

85%

### **VOA Special List**

CAS No.	Compound	Result	RL	MDL	Units	Q
67-64-1 71-43-2 100-41-4 108-10-1 108-88-3 1330-20-7	Acetone Benzene Ethylbenzene 4-Methyl-2-pentanone(MIBK) Toluene Xylene (total)	10 0.47 3830 a J 195 2.4 J 11800 a J	10 1.0 60 5.1 1.0	6.8 0.14 8.9 2.7 0.39	ug/kg ug/kg ug/kg ug/kg ug/kg ug/kg	J
CAS No.	Surrogate Recoveries  Dibromofluoromethane	Run# 1 91%	Run# 2 91%	<b>Limi</b> 67-11	ts	
17060-07-0 2037-26-5	1,2-Dichloroethane-D4 Toluene-D8	89% 95%	98% 92%	66-13 76-13		

90%

(a) Result is from Run# 2

460-00-4



ND = Not detected

MDL - Method Detection Limit RL = Reporting Limit

E = Indicates value exceeds calibration range

4-Bromofluorobenzene

J = Indicates an estimated value

B = Indicates analyte found in associated method blank

**Client Sample ID:** P-5(4.5-5.5) Lab Sample ID:

Matrix:

JA94124-12 SO - Soil

Method:

SW846-8015 (DAI)

Project:

BMS-ICM, Humacao, PR

Date Sampled: 12/08/11

Date Received: 12/09/11

Percent Solids: 83.9

File ID Run #1 GH85807.D DF 1

Analyzed 12/13/11

By XPL

**Prep Date** n/a

**Prep Batch** n/a

**Analytical Batch** GGH3921

Run #2

Initial Weight

Compound

Run #1 Run #2

CAS No.

CAS No.

5.0 g

Result RL**MDL** Units Q

67-63-0 Isopropyl Alcohol 67-56-1 Methanol

ND ND 120 45 240 61

ug/kg ug/kg

Run# 1

Run# 2

Limits 58-137%

111-27-3 Hexanol 100% 111-27-3 Hexanol 101%

**Surrogate Recoveries** 

58-137% CIADO DE tacl Infante Viende/

14 1888

Client Sample ID: FB120811 Lab Sample ID:

Matrix:

JA94124-13 AQ - Field Blank Soil

Method:

SW846 8260B

Project:

BMS-ICM, Humacao, PR

Date Sampled: 12/08/11 Date Received: 12/09/11

Percent Solids: n/a

	File ID	DF	Analyzed	$\mathbf{B}\mathbf{y}$	Prep Date	Prep Batch	<b>Analytical Batch</b>
Run #1	4B13609.D	1	12/12/11	RS	n/a	n/a	V4B588

Run #2

**Purge Volume** 

5.0 ml Run #1

Run #2

### **VOA Special List**

CAS No.	Compound	Result	RL	MDL	Units	Q
67-64-1	Acetone	ND	10	7.6	ug/l	
71-43-2	Benzene	ND	1.0	0.22	ug/l	
100-41-4	Ethylbenzene	ND	1.0	0.21	ug/l	
108-10-1	4-Methyl-2-pentanone(MIBK)	ND 3	5.0	1.2	ug/l	
108-88-3	Toluene	ND	1.0	0.15	ug/l	
1330-20-7	Xylene (total)	ND	1.0	0.17	ug/l	
CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Lim	its	
1868-53-7	Dibromofluoromethane	98%		77-1	20%	
17060-07-0	1,2-Dichloroethane-D4	91%		70-1	27%	
2037-26-5	Toluene-D8	93%		79-1	20%	
460-00-4	4-Bromofluorobenzene	85%		76-1	18%	



ND = Not detected

E = Indicates value exceeds calibration range

MDL - Method Detection Limit

J = Indicates an estimated value

B = Indicates analyte found in associated method blank

Client Sample ID: TB120811

Lab Sample ID:

JA94124-14

Matrix:

AQ - Trip Blank Soil

Method:

SW846 8260B

**Date Sampled:** 12/08/11 Date Received: 12/09/11

Percent Solids: n/a

Project:

BMS-ICM, Humacao, PR

File ID 4B13610.D Run #1

DF 1

By RS

Analyzed

12/12/11

**Prep Date** n/a

**Prep Batch** n/a

**Analytical Batch** V4B588

Run #2

**Purge Volume** 

5.0 ml

Run #1

Run #2

### **VOA Special List**

CAS No.	Compound	Result	RL	MDL	Units	Q
67-64-1	Acetone	ND	10	7.6	ug/l	
71-43-2	Benzene	ND	1.0	0.22	ug/l	
100-41-4	Ethylbenzene	ND	1.0	0.21	ug/l	
108-10-1	4-Methyl-2-pentanone(MIBK)	ND $J$	5.0	1.2	ug/l	
108-88-3	Toluene	ND	1.0	0.15	ug/l	
1330-20-7	Xylene (total)	ND	1.0	0.17	ug/l	
CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limi	its	
1868-53-7	Dibromofluoromethane	96%		77-1	20%	
17060-07-0	1,2-Dichloroethane-D4	90%		70-13	27%	
2037-26-5	Toluene-D8	92%		79-13	20%	
460-00-4	4-Bromofluorobenzene	86%		76-1	18%	



ND = Not detected

MDL - Method Detection Limit

RL = Reporting Limit

E = Indicates value exceeds calibration range

J = Indicates an estimated value

B = Indicates analyte found in associated method blank N = Indicates presumptive evidence of a compound

ACCUTEST:	SHE
LABORATORIES	519

### CHAIN OF CUSTODY

PAGE 1 OF 2

LABORATORIES	57D	Tel	2235 Ro 732-329-						490		1 8	708	259	1214	74	Bottle C	Order Control 8		
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Client / Reporting Information			Project	Inform	nation	12.000	31:04	ia n	95	-31	5 PU	Rec	queste	d Analys	sis ( see	TEST C	ODE shee		
Company Name	Project Name:	tol-Myers	r. :14	Hu	4056	DR							Г	П					DW - Orinking Water
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TerryTaylor	Project #   Buil	<u> 402 5</u>					Sta				שושבושו	-	1						AIR - Air SOL - Other Sclid
Terry Taylor - 914-251-0400 Ext. 309				City			Sta	•		Zip	7	<b>3</b>	~						WP - Wipe FB-Field Blank EB-Equipment Blank
Terry Talylor/ Nestor & Vera	Project Manag	er		Attenti	OFE						72	3	olio					1	RB-Rinse Blank TB-Trip Blank
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Accused Sample # Field ID / Point of Collection	MECHICIVES	Date	Time	Sumple by	ed Medite	# of bottles	F F	H2804	NONE DI Witter	MEOH	Ethyle XXIV	重	6						. LAB USE ONLY
1-12 (9.5-10.5)	1	12/7/11	1330	7	[ Soil	5	Ħ	Ť	2	3	×	×	×	_	_	1			
Z 1-13/12.5-13.5)		l l	1415	1	1	3		$\top$	2	3	×	<b>×</b>	×			1			FS, Z
3 I-14 (8.5-9.5)			1515	1		5		T	2	3	×	X	×						1406
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-8 I-19 (8-9)	9		1200	Ц	\Y	5	Ш	$\perp$	2	3	×	×	X			ļ			
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-10 P-9 (4.5~6)	19		1340	Н	50:1	5	Ш	_	2	3	X	×	X			$\sqcup$	$\vdash$	11	
1 P-8(4-5)		<u> </u>	1415	Ц,		5	Ш	+	2	3	X.	<u>پد</u> ا	X			Ш	$\vdash$	<del></del> _	
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e. I	2 Day EMERGENCY   1 Dey EMERGENCY   Spency & Rush T/A date available V/A Labin							Commerc	ial '8' :	Resul	s + QC	Summ	ery	at Raw dat	_		_				$\neg$		
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RAGAIRA: Chain of Custody Page 2 of 3

	Project Number:JA94124
	Date:12/07-08/2011
REVIEW OF VOLATILE ORGATH The following guidelines for evaluating volatile organics we actions. This document will assist the reviewer in using prodecision and in better serving the needs of the data users. The USEPA data validation guidance documents in the following of HW-24, Validating Volatile Organic Compounds by GC/MS, SV 2), the USEPA National Functional Guidelines for Low/Medium SOM01.2 SOP HW-33, August 2009 — Revision 2), the USEP Data Review for Low Concentration (SOP HW-13, August, 20 Methods for Evaluating Solid Waste, Physical/Chemical Methods for Evaluating Solid Waste, Physical/Chemical Methods for Evaluating Solid Waste, Physical/Chemical Methods are utilized. The Quite data review worksheets are from the primary guidance document and the quality control and performance data summare.	ere created to delineate required validation of pressional judgment to make more informed be sample results were assessed according to order of precedence: USEPA Region 2, SOP N-846 Method 8260B (August, 2009-Revision of Concentration Organic Data Review (SOW NA National Functional Guidelines for Organic 2009-Revision 3). Also, QC criteria from "Test thods SW-846 (Final Update IV, December C criteria and data validation actions listed on ument, unless otherwise noted.  data package received has been
Lab. Project/SDG No.:JA94124 No. of Samples:12	Sample matrix:Soil
Trip blank No.:  Field blank No.:  Equipment blank No.:JA94124-9  Field duplicate No.:	
X Data CompletenessX Holding TimesN/A_ GC/MS TuningN/A_ Internal Standard PerformanceX BlanksX Surrogate RecoveriesX_ Matrix Spike/Matrix Spike Duplicate	X Laboratory Control SpikesX Field DuplicatesX CalibrationsX Compound IdentificationsX Compound QuantitationX Quantitation Limits
Overall Comments:_IPA_and_Methanol_by_SW846-8015	5_(DAI)
Definition of Qualifiers:  J- Estimated results  U- Compound not detected  R- Rejected data  UJ- Estimated nordetect  Reviewer:  Date: 12/20/2011	

### **DATA REVIEW WORKSHEETS**

# DATA COMPLETENESS

MISSING INFORMATION	DATE LAB. CONTACTED	DATE RECEIVED
	-	
		•
-		

All criteria were metX
Criteria were not met
and/or see below

#### HOLDING TIMES

The objective of this parameter is to ascertain the validity of the results based on the holding time of the sample from time of collection to the time of analysis.

Complete table for all samples and note the analysis and/or preservation not within criteria

SAMPLE ID	DATE SAMPLED	DATE ANALYZED	рН	ACTION
_				
	<u> </u>			
All	samples analyzed w	ithin the recommended r	nethod h	nolding time

# **Criteria**

Aqueous samples – 14 days from sample collection for preserved samples (pH  $\leq$  2, 4°C), no air bubbles.

Aqueous samples - 7 days from sample collection for unpreserved samples, 4°C, no air bubbles.

Soil samples- 7 days from sample collection.

Cooler temperature (Criteria: 4 ± 2 °C): 4°C - OK

### Actions

If the VOCs vial(s) have air bubbles, estimate positive results (J) and reject nondetects (R).

If the % solids of soil samples is 10-50%, estimates positive results (J) and nondetects (UJ)

If the % solid of soil samples is < 10%, estimate positive results (J) and reject nondetects (R).

If holding times are exceeded but < 14 days beyond criteria, estimate positive results (J) and nondetects (UJ).

If holding times are exceeded but < 28 days beyond criteria, estimate positive results (J) and reject nondetects (R).

If holding times are grossly exceeded (> 28 days beyond criteria), reject all results (R).

If samples were not iced or if the ice were melted (> 10°C), estimate positive results (J) and nondetects (UJ).

		Crit	All criteria were metN/A eria were not met see below
GC/MS TUNING			
The assessment of standard tuning QC	•	o determine if the sample instru	mentation is within the
N/A_ The BFB pe	erformance results were	reviewed and found to be within	the specified criteria.
N/A_ BFB tuning	was performed for ever	y 12 hours of sample analysis.	
lf no, use profession		nine whether the associated dat	a should be accepted,
List	the	samples	affected:

If mass calibration is in error, all associated data are rejected.

All criteria were metX
Criteria were not met
and/or see below

# CALIBRATION VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration:11/12/11						
Dates of con	tinuing	calibrati	on:_12/13/11			
Instrument II				,		
Matrix/Level						
DATE	LAB	FILE	CRITERIA OUT	COMPOUND	SAMPLES	
	ID#		RFs, %RSD, <b>%D</b> , r		AFFECTED	
Initial and continuing calibration meet method performance criteria						
		_				

### Criteria

All RFs must be > 0.05 regardless of method requirements for SPCC.

All %RSD must be < 15 % regardless of method requirements for CCC.

All %Ds must be < 20% regardless of method requirements for CCC.

It should be noted that Region 2 SOP HW-24 does not specify criterion for the curve correlation coefficient (r). A limit for r of  $\geq$  0.995 has therefore been utilized as professional judgment.

### **Actions**

If any compound has an initial RF or a continuing RF of < 0.05, estimate positive results (J) and reject nondetects (R), regardless of method requirements.

If any compound has a %RSD > 15%, estimate positive results (J) and use professional judgment to qualify nondetects.

If any compound has a %RSD > 90%, estimate positive results (J) and reject nondetects (R).

If any compound has a % D > 20%, estimate positive results (J) and reject nondetects (R).

If any compound has a % D > 20%, estimate positive results (J) and nondetects (UJ).

If any compound has a % D > 90%, estimate positive results (J) and reject nondetects (R).

If any compound has r > 0.995, estimate positive results and nondetects.

A separate worksheet should be filled for each initial curve

All criteria were metX
Criteria were not met
and/or see below

# V A. BLANK ANALYSIS RESULTS (Sections 1 & 2)

The assessment of the blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks apply only to blanks associated with the samples, including trip, equipment, and laboratory blanks. If problems with any blanks exist, all data associated with the case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the case, or if the problem is an isolated occurrence not affecting other data.

List the contamination in the blanks below. High and low levels blanks must be treated separately.

Laboratory blanks

DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS
All_method				
Field/Equipmen  DATE  ANALYZED			COMPOUND	
	alytes_detected		nent_blank_analyzed_v	rith_this_data_package
_No_trip/equipn	nent_blanks_an	alyzed_with_th	is_data_package	
			·····	
	<del></del>	<u> </u>		

All criteria were metX
Criteria were not met
and/or see below

# VB. BLANK ANALYSIS RESULTS (Section 3)

### **Blank Actions**

Action Levels (ALs) should be based upon the highest concentration of contaminant determined in any blank. Do not qualify any blank with another blank. The ALs for samples which have been diluted should be corrected for the sample dilution factor and/or % moisture, where applicable. No positive sample results should be reported unless the concentration of the compound in the samples exceeds the ALs:

ALs = 10x the amount of common contaminants (methylene chloride, acetone, 2-butanone, and toluene)

ALs = 5x for any other compounds

Specific actions are as follows:

If the concentration is < sample quantitation limit (SQL) and  $\le$  AL, report the compound as not detected (U) at the SQL.

If the concentration is  $\geq$  SQL but  $\leq$  AL, report the compound as not detected (U) at the reported concentration.

If the concentration is  $\geq$  SQL and > AL, report the concentration unqualified.

### Notes:

High and low level blanks must be treated separately

Compounds qualified "U" for blank contamination are still considered "hits" when qualifying for calibration criteria.

CONTAMINATION SOURCE/LEVEL	COMPOUND	CONC/UNITS	AL/UNITS	SQL	AFFECTED SAMPLES
	_				
_					

All criteria were met
Criteria were not met
and/or see belowX

### SURROGATE SPIKE RECOVERIES

Laboratory performance of individual samples is established by evaluation of surrogate spike recoveries. All samples are spiked with surrogate compounds prior to sample analysis. The accuracy of the analysis is measured by the surrogate percent recovery. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the validation of data is frequently subjective and demands analytical experience and professional judgment.

List the percent recoveries (%Rs) which do not meet the criteria for surrogate recovery.

Matrix: solid/aqueous

SAMPLE ID SURROGATE COMPOUND				)	ACTION
	1,2-DCA	DBFM	TOL-d8	BFB	
JA94124-6	143 %				No_action_
_JA94124-6 _Recoveries_OK_	in signal #2;	Confirmed GC	C/MS		
_JA94124-6MS					No_action_
_JA94124-6MS	213				No_action_
_Matrix_interferen	ceRecoveries	s_OK_in_signa	al_#2;		
_SurrogateHex	ranol				
QC Limits* (Aqueo	nie)				
LL_to_UL_		to	to	to	
QC Limits* (Solid-					
		137 to	to	to	
QC Limits* (Solid-					<del></del>
LL to UL	to	to	to	to	
1,2-DCA = 1,2-Dic	hloromethane-	d4	TOL-d8	3 = Toluene-d8	
DBFM = Dibromof	luoromethane		BFB =	Bromofluoroben	zene

- \* QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- \* If QC limits are not available, use limits of 80 120 % for aqueous and 70 130 % for solid samples.

### Actions:

QUALITY	%R < 10%	%R = 10% - LL	%R > UL
Positive results	J	J	_
Nondetects results	R	UJ	Accept

Surrogate action should be applied:

If one or more surrogate in the VOC fraction is out of specification, but has a recovery of > 10%.

If any one surrogate in a fraction shows < 10 % recovery.

All criteria were metX
Criteria were not met
and/or see below

# VII. A MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

This data is generated to determine long term precision and accuracy in the analytical method for various matrices. This data alone cannot be used to evaluate the precision and accuracy of individual samples. If any % R in the MS or MSD falls outside the designated range, the reviewer should determine if there are matrix effects, i.e. LCS data are within the QC limits but MS/MSD data are outside QC limit.

# 1. MS/MSD Recoveries and Precision Criteria

The laboratory should use one MS and a duplicate analysis of an unspiked field sample if target analytes are expected in the sample. If target analytes are not expected, MS/MSD should be analyzed. List the %Rs, RPD of the compounds which do not meet the criteria.

Sample ID:JA94124-6				Matrix/Level:SOIL				
MS OR MSD	COMPOUND	% R	RPD	QC LIMITS	ACTION			
MS/MSD_recoveries_and_RPD_within_laboratory_control_limits								

- \* QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- \* If QC limits are not available, use limits of 70 130 %.

#### Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

MS/MSD criteria apply only to the unspiked sample, its dilutions, and the associated MS/MSD samples:

If the % R for the affected compounds were < LL (or 70 %), qualify positive results (J) and nondetects (UJ).

If the % R for the affected compounds were > UL (or 130 %), only qualify positive results (J).

If 25 % or more of all MS/MSD %R were < LL (or 70 %) or if two or more MS/MSD %Rs were < 10%, qualify all positive results (J) and reject nondetects (R).

A separate worksheet should be used for each MS/MSD pair.

All criteria were metX
Criteria were not met
and/or see below

# VII. B MATRIX SPIKE/MATRIX SPIKE DUPLICATE

MS/MSD - Unspiked Compounds

It should be noted that Region 2 SOP HW-24 does not specify a MS/MSD criteria for the unspiked compounds in the sample. A %RSD of < 50% has therefore been utilized as professional judgment.

If all target analytes were spiked in the MS/MSD, this review element is not applicable.

List the %RSD of the compounds which do not meet the criteria.

Sample ID:	· · · · · · · · · · · · · · · · · · ·		Matrix/Level/Unit:				
COMPOUND	SAMPLE MS CONC. CONC.				MSD CONC.	% RSD	ACTION
					_		
<u>.</u>							

# Actions:

<sup>\*</sup> If the % RSD > 50, qualify the positive result in the unspiked samples as estimated (J).

<sup>\*</sup> If the % RSD is not calculated (NC) due to nondetected value, use professional judgment to qualify the data.

All criteria were metX
Criteria were not met
and/or see below

# VIII. LABORATORY CONTROL SAMPLE (LCS) ANALYSIS

This data is generated to determine accuracy of the analytical method for various matrices.

### LCS Recoveries Criteria

Where LCS spiked with the same analyte at the same concentrations as the MS/MSD? Yes or No. If no make note in data review memo.

List the %R of compounds which do not meet the criteria

	LCS ID	COMPOUND	% R	QC LIMIT						
Recoverie	Recoveries_within_laboratory_control_limits									
	_									
	_									
				<del>- ' </del>						

- QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- \* If QC limits are not available, use limits of 70 130 %.

### Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

All analytes in the associated sample results are qualified for the following criteria.

If 25 % of the LCS recoveries were < LL (or 70 %), qualify all positive results (j) and reject nondetects (R).

If two or more LCS were below 10 %, qualify all positive results as (J) and reject nondetects (R).

# 2. Frequency Criteria:

Where LCS analyzed at the required frequency and for each matrix? <u>Yes</u> or No. If no, the data may be affected. Use professional judgment to determine the severity of the effect and qualify data accordingly. Discuss any actions below and list the samples affected.

		Criteria were not met and/or see below
IX.	FIELD DUPLICATE PRECISION	
	Sample IDs:none	Matrix:_SOIL

All critoria wore met Y

Field duplicates samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which only laboratory performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples.

The project QAPP should be reviewed for project-specific information. Suggested criteria: RPD  $\pm$  30% for aqueous samples, RPD  $\pm$  50 % for solid samples. If both samples and duplicate are <5 SQL, the RPD criteria is doubled.

COMPOUND	SQL	SAMPLE CONC.	DUPLICATE CONC.	RPD	ACTION			
	_							
No field/lah	oratory o	  unlicate analyzed a	e nart of this data nacka	na MS/N	ASD results used to			
No field/laboratory duplicate analyzed as part of this data package. MS/MSD results used to assess precision. RPD within laboratory and generally acceptable control limits								

### Actions:

Qualify as estimated positive results (J) and nondetects (UJ) for the compound that exceeded the above criteria. For organics, only the sample and duplicate will be qualified.

If an RPD cannot be calculated because one or both of the sample results is not detected, the following actions apply:

If one sample result is not detected and the other is greater than 5x the SQL qualify (J/UJ).

If one sample value is not detected and the other is greater than 5x the SQL and the SQLs for the sample and duplicate are significantly different, use professional judgment to determine if qualification is appropriate.

If one sample value is not detected and the other is less than 5x, use professional judgment to determine if qualification is appropriate.

lf	both	sample	and d	uplicate	results are	not detected,	no	action	is I	needed

All criteria were met \_\_N/A\_\_ Criteria were not met and/or see below \_\_\_\_\_

# X. INTERNAL STANDARD PERFORMANCE

The assessment of the internal standard (IS) parameter is used to assist the data reviewer in determining the condition of the analytical instrumentation.

List the internal standard area of samples which do not meet the criteria.

- \* Area of +100% or -50% of the IS area in the associated calibration standard.
- \* Retention time (RT) within 30 seconds of the IS area in the associated calibration standard.

DATE	SAMPLE ID	IS OUT	IS AREA	ACCEPTABLE ACTION RANGE
	· · · · · · · · · · · · · · · · · · ·			
			•	
				<u></u>
		_		
	-	,		

Actions:

1. IS actions should be applied to the compound quantitated with the out-of-control ISs

QUALITY	IS AREA < -25%	IS AREA = -25 % TO - 50%	IS AREA > + 100%
Positive results	J	J	J
Nondetected results	R	UJ	ACCEPT

If a IS retention time varies more than 30 seconds, the chromatographic profile for that sample must be examined to determine if any false positive or negative exists. For shifts of a large magnitude, the reviewer may consider partial or total rejection of the data for the sample fraction.

All criteria were metX
Criteria were not met
and/or see below

# XII. SAMPLE QUANTITATION

The sample quantitation evaluation is to verify laboratory quantitation results. In the space below, please show a minimum of one sample calculation:

JA94124-2

Methanol

RF = 13.07

[] = (10921)/(13.07)

= 835,6 ppb OK

More than 40 % RPD for detected Isopropyl Alcohol concentrations between the two GC columns in samples JA94124-1. Results qualified as estimated (J).

All criteria were met _X
Criteria were not met
and/or see below

XII.		NITIT	$M = M \cap M$	LIMITS
AII.	<b>WUH</b>	(N 1 1 1 1	HIUN	LIMILIO

# A. Dilution performed

SAMPLE ID	DILUTION FACTOR	REASON FOR DILUTION
	-	
	· · · · · · · · · · · · · · · · · · ·	

3.	Percent Solids		
	List samples which have	≤ 50 % solids	
Actions			
	If the % solids of a soil sa	ample is 10-50%, estimate positiv	re results (J) and nondetects (UJ)
	If the % solids of a soil sa (R)	ample is < 10%, estimate positive	e results (J) and reject nondetects

	Project Number:JA94124
	Date:12/07-08/2011
REVIEW OF VOLATILE ORGA The following guidelines for evaluating volatile organics we	ere created to delineate required validation
actions. This document will assist the reviewer in using prodecision and in better serving the needs of the data users. The USEPA data validation guidance documents in the following of HW-24, Validating Volatile Organic Compounds by GC/MS, SV 2), the USEPA National Functional Guidelines for Low/Medium SOM01.2 SOP HW-33, August 2009 – Revision 2), the USEP Data Review for Low Concentration (SOP HW-13, August, 20	e sample results were assessed according to brder of precedence: USEPA Region 2, SOF W-846 Method 8260B (August, 2009-Revision Concentration Organic Data Review (SOV PA National Functional Guidelines for Organic
Methods for Evaluating Solid Waste, Physical/Chemical Me 1998)," specifically for Methods 8000/8260B are utilized. The on the data review worksheets are from the primary guidance of The hardcopied (laboratory name) _Accutest reviewed and the quality control and performance data summa	ethods SW-846 (Final Update IV, December QC criteria and data validation actions listed document, unless otherwise noted.  data package received has been
Lab. Project/SDG No.:JA94124 No. of Samples:14	
Trip blank No.:JA94124-14	
X Data Completeness X Holding Times	X Laboratory Control Spikes X Field Duplicates
X GC/MS Tuning	X Calibrations
X Internal Standard PerformanceX Blanks	X Compound Identifications X Compound Quantitation
X Surrogate Recoveries X Matrix Spike/Matrix Spike Duplicate	X Quantitation Limits
Overall Comments:_Selected_VOC's_by_SW846-8260B_	
Definition of Qualifiers:	
J- Estimated results U- Compound not detected	
R- Rejected data	
UJ- Estimated nondefect	
Reviewer: 12/22/2011	

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# DATA COMPLETENESS

MISSING INFORMATION	DATE LAB. CONTACTED	DATE RECEIVED
	_ <del></del>	
		_

All criteria were metX
Criteria were not met
and/or see below

#### HOLDING TIMES

The objective of this parameter is to ascertain the validity of the results based on the holding time of the sample from time of collection to the time of analysis.

Complete table for all samples and note the analysis and/or preservation not within criteria

SAMPLE ID	DATE SAMPLED	DATE ANALYZED	pН	ACTION
			<u> </u>	
All	samples analyzed w	ithin the recommended r	nethod h	olding time
		_		
				-
			_	
		, _		

# <u>Criteria</u>

Aqueous samples – 14 days from sample collection for preserved samples (pH  $\leq$  2, 4°C), no air bubbles.

Aqueous samples – 7 days from sample collection for unpreserved samples, 4°C, no air bubbles.

Soil samples- 7 days from sample collection.

Cooler temperature (Criteria: 4 + 2 °C): 4°C - OK

# **Actions**

If the VOCs vial(s) have air bubbles, estimate positive results (J) and reject nondetects (R).

If the % solids of soil samples is 10-50%, estimates positive results (J) and nondetects (UJ)

If the % solid of soil samples is < 10%, estimate positive results (J) and reject nondetects (R).

If holding times are exceeded but < 14 days beyond criteria, estimate positive results (J) and nondetects (UJ).

If holding times are exceeded but < 28 days beyond criteria, estimate positive results (J) and reject nondetects (R).

If holding times are grossly exceeded (> 28 days beyond criteria), reject all results (R).

If samples were not iced or if the ice were melted (> 10°C), estimate positive results (J) and nondetects (UJ).

		Crit	All criteria were metX teria were not met see below
GC/MS TUNING			
The assessment of standard tuning QC	_	o determine if the sample instru	mentation is within the
XThe BFB p	erformance results were	reviewed and found to be within	the specified criteria.
XBFB tuning	g was performed for ever	y 12 hours of sample analysis.	
lf no, use professi qualified or rejected		nine whether the associated da	ta should be accepted,
List	the	samples	affected:

All criteria were met
Criteria were not met
and/or see belowX

### CALIBRATION VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration:	_09/14/11	_11/12/11	11/08/11
Dates of continuing calibration:	12/12/11	12/14/11	12/10/11
Instrument ID numbers:	GCMS4B	GCMSE	GCMSY
Matrix/Level:Aqueous/low			
•			

DATE	LAB FILE ID#	CRITERIA OUT RFs, %RSD, <b>%D</b> , r	COMPOUND	SAMPLES AFFECTED
12/08/2011	cc435-20	-23.6	MIBK	JA94124-9;-13;-14
				_

### Criteria

All RFs must be > 0.05 regardless of method requirements for SPCC.

All %RSD must be < 15 % regardless of method requirements for CCC.

All %Ds must be < 20% regardless of method requirements for CCC.

It should be noted that Region 2 SOP HW-24 does not specify criterion for the curve correlation coefficient (r). A limit for r of  $\geq$  0.995 has therefore been utilized as professional judgment.

### **Actions**

If any compound has an initial RF or a continuing RF of < 0.05, estimate positive results (J) and reject nondetects (R), regardless of method requirements.

If any compound has a %RSD > 15%, estimate positive results (J) and use professional judgment to qualify nondetects.

If any compound has a %RSD > 90%, estimate positive results (J) and reject nondetects (R).

If any compound has a % D > 20%, estimate positive results (J) and reject nondetects (R).

If any compound has a % D > 20%, estimate positive results (J) and nondetects (UJ).

If any compound has a % D > 90%, estimate positive results (J) and reject nondetects (R).

If any compound has r > 0.995, estimate positive results and nondetects.

A separate worksheet should be filled for each initial curve

All criteria were metX
Criteria were not met
and/or see below

# V A. BLANK ANALYSIS RESULTS (Sections 1 & 2)

The assessment of the blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks apply only to blanks associated with the samples, including trip, equipment, and laboratory blanks. If problems with any blanks exist, all data associated with the case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the case, or if the problem is an isolated occurrence not affecting other data.

List the contamination in the blanks below. High and low levels blanks must be treated separately.

Laboratory blanks

DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS
DATE ANALYZED	LAB ID	LEVEL/ Matrix	COMPOUND	CONCENTRATION UNITS
_No_target_ana package	•			nalyzed_with_this_data

All criteria were metX
Criteria were not met
and/or see below

# VB. BLANK ANALYSIS RESULTS (Section 3)

# **Blank Actions**

Action Levels (ALs) should be based upon the highest concentration of contaminant determined in any blank. Do not qualify any blank with another blank. The ALs for samples which have been diluted should be corrected for the sample dilution factor and/or % moisture, where applicable. No positive sample results should be reported unless the concentration of the compound in the samples exceeds the ALs:

ALs = 10x the amount of common contaminants (methylene chloride, acetone, 2-butanone, and toluene)

ALs = 5x for any other compounds

Specific actions are as follows:

If the concentration is < sample quantitation limit (SQL) and  $\le$  AL, report the compound as not detected (U) at the SQL.

If the concentration is  $\geq$  SQL but  $\leq$  AL, report the compound as not detected (U) at the reported concentration.

If the concentration is > SQL and > AL, report the concentration unqualified.

# Notes:

High and low level blanks must be treated separately

Compounds qualified "U" for blank contamination are still considered "hits" when qualifying for calibration criteria.

CONTAMINATION SOURCE/LEVEL	COMPOUND	CONC/UNITS	AL/UNITS	SQL	AFFECTED SAMPLES
_					
				<u> </u>	
		ļ			
	!				

All criteria were met _X
Criteria were not met
and/or see below

### SURROGATE SPIKE RECOVERIES

Laboratory performance of individual samples is established by evaluation of surrogate spike recoveries. All samples are spiked with surrogate compounds prior to sample analysis. The accuracy of the analysis is measured by the surrogate percent recovery. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the validation of data is frequently subjective and demands analytical experience and professional judgment.

List the percent recoveries (%Rs) which do not meet the criteria for surrogate recovery. Matrix: solid/aqueous

SAMPLE ID	1,2-DCA	DBFM	TOL-d8	BFB	ACTION	
_All_surrogate_reco	veries_withir	_laboratory_co	ontrol_limits			
		<del></del>			<u> </u>	
		· · · · · · · · · · · · · · · · · · ·				
QC Limits* (Aqueous	s)					
LL_to_UL	to_	to_	to	to		
QC Limits* (Solid-Lo	w)					
LL_to_UL	to_	to_	to	to	<del></del>	
QC Limits* (Solid-Me	ed)					
LL_to_UL	to	to	to	to	<del></del>	
1,2-DCA = 1,2-Dichle	oromethane-	d4	TOL-d	8 = Toluene-d8		
DBFM = Dibromofluc	oromethane		BFB =	Bromofluorober	nzene	

- \* QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- \* If QC limits are not available, use limits of 80 120 % for aqueous and 70 130 % for solid samples.

### Actions:

QUALITY	%R < 10%	%R = 10% - LL	%R > UL
Positive results	J	J	J
Nondetects results	R	UJ	Accept

Surrogate action should be applied:

If one or more surrogate in the VOC fraction is out of specification, but has a recovery of > 10%. If any one surrogate in a fraction shows < 10 % recovery.

All criteria were met
Criteria were not met
and/or see belowX

# VII. A MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

This data is generated to determine long term precision and accuracy in the analytical method for various matrices. This data alone cannot be used to evaluate the precision and accuracy of individual samples. If any % R in the MS or MSD falls outside the designated range, the reviewer should determine if there are matrix effects, i.e. LCS data are within the QC limits but MS/MSD data are outside QC limit.

# 1. MS/MSD Recoveries and Precision Criteria

The laboratory should use one MS and a duplicate analysis of an unspiked field sample if target analytes are expected in the sample. If target analytes are not expected, MS/MSD should be analyzed. List the %Rs, RPD of the compounds which do not meet the criteria.

Sample ID:JA Sample ID:JA Sample ID:JA	\94124-3		Matrix	/Level:AQUEC /Level:SOIL /Level:SOIL	
MS OR MSD JA94124-5	COMPOUND	% R	RPD	QC LIMITS	ACTION
MS	Ethylbenzene	172		20144	Qualify_results_(J)_
MS	Toluene	146_		29138	affected_samples
MS	Xylenes(Total)	176_		18145	

<sup>\*</sup> QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.

### Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

MS/MSD criteria apply only to the unspiked sample, its dilutions, and the associated MS/MSD samples:

If the % R for the affected compounds were < LL (or 70 %), qualify positive results (J) and nondetects (UJ).

If the % R for the affected compounds were > UL (or 130 %), only qualify positive results (J).

If 25 % or more of all MS/MSD %R were < LL (or 70 %) or if two or more MS/MSD %Rs were < 10%, qualify all positive results (J) and reject nondetects (R).

A separate worksheet should be used for each MS/MSD pair.

<sup>\*</sup> If QC limits are not available, use limits of 70 – 130 %.

All criteria were metX
Criteria were not met
and/or see below

# VII. B MATRIX SPIKE/MATRIX SPIKE DUPLICATE

MS/MSD - Unspiked Compounds

It should be noted that Region 2 SOP HW-24 does not specify a MS/MSD criteria for the unspiked compounds in the sample. A %RSD of < 50% has therefore been utilized as professional judgment.

If all target analytes were spiked in the MS/MSD, this review element is not applicable.

List the %RSD of the compounds which do not meet the criteria.

Sample ID:			Matrix/Level/Unit:		
COMPOUND	SAMPLE CONC.	MS CONC.	MSD CONC.	% RSD	ACTION
					_

# Actions:

<sup>\*</sup> If the % RSD > 50, qualify the positive result in the unspiked samples as estimated (J).

<sup>\*</sup> If the % RSD is not calculated (NC) due to nondetected value, use professional judgment to qualify the data.

All criteria were metX
Criteria were not met
and/or see below

# VIII. LABORATORY CONTROL SAMPLE (LCS) ANALYSIS

This data is generated to determine accuracy of the analytical method for various matrices.

1. LCS Recoveries Criteria

Where LCS spiked with the same analyte at the same concentrations as the MS/MSD? Yes or No. If no make note in data review memo.

List the %R of compounds which do not meet the criteria

	LCS ID	COMPOUND	% K	QC LIMIT			
Recoverie	Recoveries_within_laboratory_control_limits						

- \* QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- \* If QC limits are not available, use limits of 70 130 %.

#### Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

All analytes in the associated sample results are qualified for the following criteria.

If 25 % of the LCS recoveries were < LL (or 70 %), qualify all positive results (j) and reject nondetects (R).

If two or more LCS were below 10 %, qualify all positive results as (J) and reject nondetects (R).

# 2. Frequency Criteria:

Where LCS analyzed at the required frequency and for each matrix? <u>Yes</u> or No. If no, the data may be affected. Use professional judgment to determine the severity of the effect and qualify data accordingly. Discuss any actions below and list the samples affected.

All criteria were metX
Criteria were not met
and/or see below

# IX. LABORATORY DUPLICATE PRECISION

Sample IDs:	JA94124-4	Matrix:_SOIL

Field duplicates samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which only laboratory performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples.

The project QAPP should be reviewed for project-specific information. Suggested criteria: RPD  $\pm$  30% for aqueous samples, RPD  $\pm$  50 % for solid samples. If both samples and duplicate are <5 SQL, the RPD criteria is doubled.

COMPOUND	SQL	SAMPLE CONC.	DUPLICATE CONC.	RPD	ACTION	
Benzene	0.14	0.76	0.31	84	Qualify results (J) in	
Ethylbenzene	0.15	0.38	ND	200	sample JA94124	
Xylene(total)	0.19	7.5	2.0	116		
Note: laboratory states that "High RPD due to possible sample analyzed from different vials";						
therefore results qualified based on professional judgment						

#### Actions:

Qualify as estimated positive results (J) and nondetects (UJ) for the compound that exceeded the above criteria. For organics, only the sample and duplicate will be qualified.

If an RPD cannot be calculated because one or both of the sample results is not detected, the following actions apply:

If one sample result is not detected and the other is greater than 5x the SQL qualify (J/UJ).

If one sample value is not detected and the other is greater than 5x the SQL and the SQLs for the sample and duplicate are significantly different, use professional judgment to determine if qualification is appropriate.

If one sample value is not detected and the other is less than 5x, use professional judgment to determine if qualification is appropriate.

If both sample and duplicate results are not detected, no action is needed.

All criteria were metX
Criteria were not met
and/or see below

# X. INTERNAL STANDARD PERFORMANCE

The assessment of the internal standard (IS) parameter is used to assist the data reviewer in determining the condition of the analytical instrumentation.

List the internal standard area of samples which do not meet the criteria.

- \* Area of +100% or -50% of the IS area in the associated calibration standard.
- \* Retention time (RT) within 30 seconds of the IS area in the associated calibration standard.

DATE	SAMPLE ID	IS OUT	IS AREA	ACCEPTABLE RANGE	ACTION
_Internal_s	tandard_area_within_	_laboratory_cor	trol_limits		
Actions:	<del>, , , , , , , , , , , , , , , , , , , </del>		17.00		

1. IS actions should be applied to the compound quantitated with the out-of-control ISs

QUALITY	IS AREA < -25%	IS AREA = -25 %	IS AREA > + 100%
		TO – 50%	
Positive results	J	J	J
Nondetected results	R	UJ	ACCEPT

If a IS retention time varies more than 30 seconds, the chromatographic profile for that sample must be examined to determine if any false positive or negative exists. For shifts of a large magnitude, the reviewer may consider partial or total rejection of the data for the sample fraction.

All criteria were met \_\_X\_\_ Criteria were not met and/or see below \_\_\_\_

# XII. SAMPLE QUANTITATION

The sample quantitation evaluation is to verify laboratory quantitation results. In the space below, please show a minimum of one sample calculation:

JA94124-1

**ACETONE** 

RF = 0.071

[] = (231223)(50)/(168133)(0.071)

= 968.5 ppb OK

All criteria were met _X
Criteria were not met
and/or see below

# XII. QUANTITATION LIMITS

# A. Dilution performed

CAMPLEID	DILLITION EACTOR	DEACON FOR DILLITION
SAMPLE ID	DILUTION FACTOR	REASON FOR DILUTION
JA94124-6	10 x	Ethylbenzene and total xylenes outside calibration range
JA94124-10	10 x	MIBK and total xylenes outside calibration range

Percent Solids		
List samples which have	≤50 % solids	
If the % solids of a soil sa	ample is 10-50%, estimate po	ositive results (J) and nondetects (UJ)
If the % solids of a soil sa (R)	ample is < 10%, estimate pos	sitive results (J) and reject nondetects
	If the % solids of a soil sa	List samples which have ≤ 50 % solids  If the % solids of a soil sample is 10-50%, estimate positive with the % solids of a soil sample is < 10%, estimate positive with the % solids of a soil sample is < 10%, estimate positive with the % solids of a soil sample is < 10%, estimate positive with the % solids of a soil sample is < 10%, estimate positive with the % solids of a soil sample is < 10%, estimate positive with the % solids of a soil sample is < 10%, estimate positive with the % solids of a soil sample is < 10%, estimate positive with the % solids of a soil sample is < 10%, estimate positive with the % solids of a soil sample is < 10%, estimate positive with the % solids of a soil sample is < 10%, estimate positive with the % solids of a soil sample is < 10%, estimate positive with the % solids of a soil sample is < 10%, estimate positive with the % solids of a soil sample is < 10%, estimate positive with the % solids of a soil sample is < 10%, estimate positive with the % solids of a soil sample is < 10%, estimate positive with the % solids of a soil sample is < 10%, estimate positive with the % solids of a soil sample is < 10%, estimate positive with the % solids of a soil sample is < 10%, estimate positive with the % solids of a soil sample is < 10%.